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(19) (CA) **CANADIAN PATENT** (12)

(54) Process for the Treatment of Iron- and Zinc-
Containing Hydrochloric Acid Pickles

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Germany (Federal Republic of)

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(33 38 258.1) 831021

No. OF CLAIMS 11

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ABSTRACT

The invention relates to a process for the treatment of iron- and zinc-containing spent hydrochloric acid solutions by liquid-liquid extraction wherein for the separation of iron and zinc the iron present in the pickles in bi- and trivalent form is converted completely to bivalent iron through a preceding reduction, the zinc is extracted with organic solvents containing complexing agents, and the phases containing iron and zinc are treated conventionally after reextraction. The trivalent iron is then reduced to the +2 oxidation state in two stages through successive additions of metallic iron and zinc.

In the packed column 14, the iron(II) chloride solution is oxidized by the admission of gaseous oxidizing agents to iron(III) chloride solution, which can be used as a flocculant, for example, in water conditioning.

Practical example

A spent hydrochloric acid having the following composition, for example, was used:

Free HCl	39.7 g/l
Fe ⁺²	107.4 g/l
Fe ⁺³	6.6 g/l
Zn	47.9 g/l
Pb	403.0 mg/l
Sn	51.8 mg/l
Cd	38.2 mg/l

Surface tension: 0.062 N/m.

Treatment in the two-stage cementation unit resulted in the following values:

Free HCl	0.05 g/l
Fe ⁺²	144.3 g/l
Fe ⁺³	Not detectable
Zn	47.7 g/l
Pb	3.2 mg/l
Sn	2.0 mg/l
Cd	1.3 mg/l

Surface tension: 0.083 N/m.

This solution was fed to a three-stage extraction unit followed by a fixed-bed anion exchanger, at the exit of which it had a residual zinc content of 0.6 mg/l. The zinc-laden organic phase was extracted with water. The raffinate containing zinc chloride so obtained contained 23.8 g/l zinc.

THE EMBODIMENTS OF THE INVENTION IN WHICH AN EXCLUSIVE PROPERTY OR PRIVILEGE IS CLAIMED ARE DEFINED AS FOLLOWS:

1. A process for the treatment of iron- and zinc-containing spent hydrochloric acid solutions by liquid-liquid extraction wherein for the separation of iron and zinc the iron present in the pickles in bi- and trivalent form is converted completely to bivalent iron through a preceding reduction, the zinc is extracted with organic solvents containing complexing agents, and the phases containing iron and zinc are treated conventionally after reextraction, the reduction of the trivalent iron to the +2 oxidation state being effected in two stages through successive additions of metallic iron and zinc.

2. A process according to claim 1, in which that the addition of iron and zinc is carried out so that in a first stage a pH value of about 3 is established by the controlled addition of metallic iron, and in a second stage metallic zinc is added in an amount sufficient for the precipitation of any heavy metal still present in the solution.

3. A process according to claim 1, in which reduction of the trivalent iron and the extraction stage are carried out under an inert gas.

4. A process according to claims 1, 2 or 3, in which prior to said treatment the spent acid solutions are treated with adsorbents.

5. A process according to claim 1, in which the extraction stage is followed by a wash of the organic phase with a dilute zinc chloride solution.

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6. A process according to claim 5, in which the zinc chloride solution used is recirculated and regenerated by precipitation with zinc oxide of the iron taken up.

7. A process for the treatment of a spent hydrochloric acid solution containing dissolved iron and zinc, which process comprises contacting said solution with metallic iron in a first stage such that trivalent iron present in the solution is reduced to divalent iron and whereby a pH value of about 3 is established, next, in a second stage contacting said solution containing dissolved zinc and divalent iron with metallic zinc, and then extracting zinc, as zinc chloride, from the resulting aqueous phase with an organic liquid containing a complexing agent for zinc chloride.

8. A process as in claim 3, wherein said reduction of trivalent iron and said extraction are carried out under an inert gas.

9. A process as in claim 7, wherein said spent acid solutions are treated with adsorbents prior to said first stage.

10. A process as in claim 7, wherein, following extraction, the organic liquid is washed with a dilute zinc chloride solution.

11. A process as in claim 10, wherein said dilute zinc chloride wash solution is recirculated and is regenerated by adding zinc oxide thereto, whereby iron taken up into said wash solution is precipitated as iron hydroxide.

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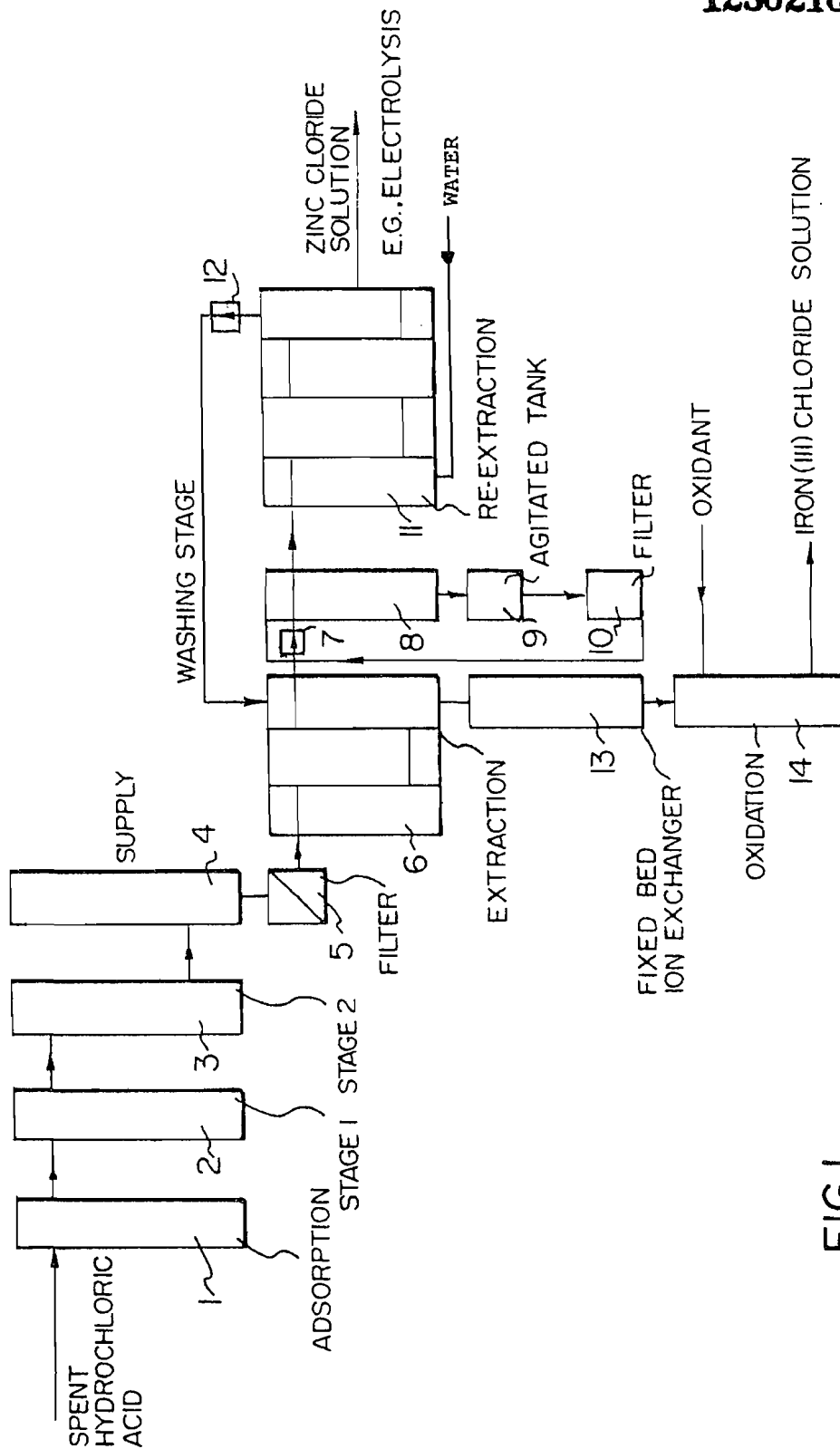


FIG. 1

Marks & Clerk

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(12) **Patent:**

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(21) **465152**

(54) PROCESS FOR THE TREATMENT OF IRON- AND ZINC- CONTAINING
HYDROCHLORIC ACID PICKLES

(54) PROCEDE DE TRAITEMENT DE BAINS D'ACIDE CHLOROHYDRIQUE
CONTENANT DU FER ET DU ZINC

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The invention relates to a process for the treatment of iron- and zinc-containing spent hydrochloric acid solutions by liquid-liquid extraction wherein for the separation of iron and zinc the iron present in the pickles in bi- and trivalent form is converted completely to bivalent iron through a preceding reduction, the zinc is extracted with organic solvents containing complexing agents, and the phases containing iron and zinc are treated conventionally after reextraction. The trivalent iron is then reduced to the +2 oxidation state in two stages through successive additions of metallic iron and zinc.

CLAIMS: [Show all claims](#)

*** Note: Data on abstracts and claims is shown in the official language in which it was submitted.

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